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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

KOBAYASHI, Yoshihiro, et al.

Serial No: 09/201,260

Filed: November 30, 1998

For: ZIRCONIUM CERAMIC  
MATERIAL, OPTICAL FIBER  
CONNECTOR MEMBER USING  
THE SAME, AND METHOD OF  
PRODUCING THE SAME

Art Unit: 2877

Examiner: Stafira, M.

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Dear Sir:

Enclosed herewith is a certified copy of a translation of Japanese Patent  
Application No. 328973/1997 which was filed November 28, 1997, from which  
priority is claimed under 35 U.S.C. § 119 and Rule 55.

Acknowledgment of the priority document is respectfully requested to ensure  
that the subject information appears on the printed patent.

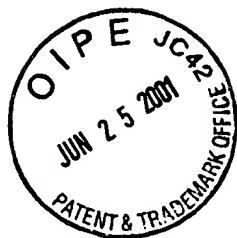
Respectfully submitted,

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**This is to certify that the annexed is a true copy of the following application as filed with this Office.**

**Date of Application:** November 28, 1997  
**Application Number:** 328973/1997  
**Applicant(s):** KYOCERA CORPORATION

**October 30, 1998**

**Commissioner,  
Patent Office**

**Takeshi ISAYAMA  
(seal)**

**Document Name:** Application for Patent  
**Docket No.:** 17555  
**Date of Application:** November 28, 1997  
**Addressee:** Commissioner, Patent Office  
**International Patent Classification:** G02B 6/24  
**Title of the Invention:** A FERRULE FOR AN OPTICAL FIBER CONNECTOR  
**Number of Claims:** 2

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**Payment of Fees:**

**Prepayment Book No.:** 005337  
**Amount to be paid:** ¥21,000

**Attached document:**

<b>Item:</b>	<b>Specification</b>	<b>1 copy</b>
<b>Item:</b>	<b>Drawings</b>	<b>1 copy</b>

**Item:**

**Abstract**

**1 copy**

**Proof:**

**Yes**

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[Document] specification  
[title of the Invention] a ferrule for an optical fiber connector  
[Claims]

[claim 1] A ferrule for an optical fiber connector comprising a ceramic material, wherein the ceramic material has 0.1 wt% or less of a rate of change in weight while the ceramic material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours.

[Claim 2] The ferrule according to Claim 1, wherein the ceramic material comprises a zirconium ceramic including a stabilizer other than  $Y_2O_3$ .

[detailed description of the invention]

[0001]

[Field of the Invention]

The present invention relates to a ferrule for holding an optical fiber in a connector for connecting an optical fiber to another optical fiber or any of various optical devices.

[0002]

[Prior Art]

Recently, there have been widely performed communications using optical fibers. In this optical communication field, optical fiber connectors are used to connect an optical fiber to another optical fiber or any other various kinds of optical devices.

[0003]

For the optical connector member, as shown in Figs. 1, a ferrule 1 is used which has a through hole along the axis thereof, and a rear end of the ferrule is held in the backing body 2. The optical fiber 3 is inserted through the hole 1a and adhered to the ferrule. The end surfaces 1b of a pair of ferrules with the fibers are abutted to each other to form an optical fiber connector. In some cases, a ferrule 1 is connected to a package which contains an optical device to optically connect the fiber to the optical device.

[0004]

For ferrule 1, metal has been utilized, but at present the ceramics is used to be formed with high precision and to improve wear resistance. Particularly, zirconium based ceramics partially stabilized with  $Y_2O_3$  are widely used for the purpose because of properties of high strength and the like. Also, various plastics are considered to form ferrules at lower cost.

[0005]

[Problems to be solved by the Invention]

The application ranges of the optical fiber connectors have been

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recently expanded so that the connectors are required to exhibit more durability for using as optical fiber connectors. For example, optical fiber connectors, have been located for a long time in severe circumstances at high temperature and humidity, such as in deserts or abysses and the materials for the connectors is required to be usable in a longtime steady state without being exchanged.

[0006]

However,  $Y_2O_3$ -partially-stabilized zirconia-based ceramics which have widely used have a problem of reducing its properties while exposed in high temperature and humidity circumstances by transforming its tetragonal crystal to monoclinic.

[0007]

[Means to Solve the Problems]

The present invention provides a ferrule having a through hole to fix an optical fiber therein, wherein the ferrule is formed of a ceramic material having 0.1 wt% or less of a rate of change in weight before and after the material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours.

[0008]

From the results of various experiments it is found that the weight change of ceramic before and after holding in the test conducted at high temperature and high humidity is highly related to durability property of the material, and in the invention, the high durability in the high-temperature and high-humidity circumstances may be obtained by using materials capable of lowering the weight change to 0.1% or less.

[0009]

In generally, the term ceramic in the present invention means the material to be obtained by subjecting an inorganic matter to a high-temperature treatment, and may include single crystal, glass and composite material thereof, as well as fine ceramic.

[0010]

Further, the present invention is characterized in that the ceramic material comprises a zirconium ceramic including a stabilizer other than  $Y_2O_3$ .

[0011]

[Embodiments of the Invention]

The embodiments of the present invention will be described in detail below.

[0012]

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Fig. 1 shows a ferrule 1 in the invention, which is a cylindrical body having a through hole 1a, and in the case of fabricating an optical fiber connector, the ferrule is provided with a backing body 2 on the outer periphery with an optical fiber 3 being inserted into the through hole 1a and fixed thereto by an adhesive. Then, a pair of the ferrules 1 are fabricated to an optical fiber connector by abutting the optical end surfaces 1b of the fibers in the ferrules. In another way, the ferrule may be connected to a package containing an optical device.

[0013]

In the invention, the ferrule 1 uses a ceramic material characterized by 0.1 wt% or less of a rate of change in weight before and after the material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours.

[0014]

In the present invention, the change in weight in the above circumstances is noted to estimate the material in high-temperature and high-humidity durability. The inventor was found that the use of the material having the weight change rate of 0.1 wt% or less does not increase the connection optical loss for a long time.

[0015]

For example,  $Y_2O_3$ -partially-stabilized zirconia-based ceramics above mentioned are easily transformed from its tetragonal crystal to monoclinic during experiments at high temperature and high humidity, and hydrated on the surface layer of the material to loss its weight. Such materials which are apt to form hydrated layers on the surface decrease properties of the material in the high-temperature and high-humidity circumstances, resulting in lower durability.

[0016]

If the material which makes up ferrules 1 and is much porous or hygroscopic, it would change in weight during the high-temperature and high humidity experiments by impregnating water. Such a ferrule material having absorbed water attaches the water to an optical fiber, which water decreases the strength of the fiber, and in the worst cases, results in breaking down the fiber.

[0017]

Thus, the measurement of the weight change rate during the high-temperature and high-humidity experiments is useful to detect the presence or absence of hydrated surface layers and hygroscopic property, and is useful to estimate the duration in use under the high-temperature and

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high-humidity circumstances. Further, the material not to absorb water and difficult to be hydrated can increase duration of the ferrule 1 in use under the high-temperature and high-humidity conditions.

[0018]

Ceramic in the present invention means the general material to be obtained by treating an inorganic matter at high temperature, and includes single crystal, glass and composite material thereof as well as fine ceramic. The embodiments will be described below.

[0019]

The ceramics in the embodiment of the present invention includes Zirconia-based ceramics which is stabilized by  $\text{MgO}$ ,  $\text{CeO}_2$ ,  $\text{Dy}_2\text{O}_3$ , or  $\text{CaO}$  or these two or more oxides as stabilizing agent, without substantially containing  $\text{Y}_2\text{O}_3$ . The zirconia-based ceramics containing the stabilizing agent except  $\text{Y}_2\text{O}_3$  are hardly hydrated on the surfaces and can be superior in durability in the high temperature and humidity circumstances.

[0020]

$\text{MgO}$  or  $\text{CeO}_2$ - $\text{Dy}_2\text{O}_3$  is preferably adopted as stabilizing agent for the zirconia-based ceramics. Particularly, such sintered ceramic material may comprise  $\text{MgO}$  in a amount of 3.0-3.8 wt%, and  $\text{ZrO}_2$  as a main component, and may contain 10-40 mol% of monoclinic zirconia crystals. This ceramic material is high in heat resistance, having higher transverse rupture strength than  $7000 \text{ kg/cm}^2$ , higher fracture toughness  $K_{IC}$  than  $11 \text{ MPa}\sqrt{\text{m}}$  and higher heat-shock resistance  $\Delta T$  than  $400^\circ\text{C}$ .

[0021]

Furthermore, another sintered ceramic in the embodiment may comprise 0.5-4.5 mol% of  $\text{Dy}_2\text{O}_3$  and 2 - 8 mol% of  $\text{CeO}_2$ , in which 6 mol% or more of sum of  $\text{Dy}_2\text{O}_3$  and  $\text{CeO}_2$  is contained, and  $\text{ZrO}_2$  as a main component, and contains 50 mol% or less of monoclinic zirconia crystal. This ceramics material can have not only higher transverse rupture strength than  $5500 \text{ kg/cm}^2$  but also high heat resistance and water resistance, so that it can be avoided from deteriorating even in the high temperature and humidity circumstances.

[0022]

The ceramics may have a structure of mean grain size of  $2 \mu\text{m}$  or less and a crystal fraction of 40 mol% or less of monoclinic crystal. For another ceramics, ceramics having main ingredient of  $\text{Al}_2\text{O}_3$  in mixture with zirconia, and ceramics having main ingredient of  $\text{Al}_2\text{O}_3$  in mixture with  $\text{Al}_2\text{B}_2\text{O}_7$ , may be used.

[0023]



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The  $\text{ZrO}_2$ -dispersed  $\text{Al}_2\text{O}_3$  based ceramic is formed to disperse a predetermined amount of  $\text{ZrO}_2$  in alumina ceramic, then, to prevent cracks present as rupture sources in the ceramics from developing to rupture, thereby increasing the strength. Particularly, the  $\text{ZrO}_2$ -dispersed alumina-based ceramics may be used, in which the micro-particles of the zirconia in nanometer sizes are dispersed in grain boundaries of the alumina structure to enhance the strength of grain boundary.

[0024]

The ceramics may be selected from the silicon carbide, silicon nitride, aluminum nitride, steatite, titania, forsterite, cordierite and mulite, and sapphire as single crystal of alumina, and sintered composites comprising a ceramics component and a metal component may be used.

[0025]

The ceramics components may be used which include alumina, silicon carbide or silicon nitride and the metal components of aluminum alloys. As an example, a composite material of silicon carbide and aluminum alloy may be formed by steps of preparing a SiC-gel  $\text{SiO}_2$  mixture, pressing the mixture to compacts in a desired shape, then firing the compacts at  $800^\circ\text{C}$  to be sintered, and impregnating molten aluminum to the sintered ceramic body using a pressure-casting technique.

[0026]

Glass ceramics may also be used for the optical connector members, including borosilicate glass and crystallized glass. The crystallized glass may be formed by crystallizing  $\text{Li}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2$  system glass.

[0027]

The ceramic composites can be provided with some properties which cannot be obtained from only each component material to compose the composite, which usually comprises a matrix material and any of dispersing or reinforcing agent.

[0028]

For example, the above ceramics may be used as a matrix material to which dispersing or reinforcing agent such as other ceramic, metal, is added. Alternatively, the above ceramics may be used as a dispersing or reinforcing agent to be added in a matrix of said other ceramic or metal, or resin.

[0029]

The various kinds of ceramics mentioned above can be used for ferrule material by adjusting its composition, crystal structure, grain size

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and pore size so as to meet the weight change rate of 0.1% or less after the material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours.

[0030]

In a method of producing a ferrule for optical connector members, tubes with a through hole are formed by extruding or injection molding a slip of the material powder which is prepared in the above composition, and fired at a predetermined temperature to obtain sintered tubes. The peripheral and the hole of each tube are machined to obtain a ferrule with the desired dimensions by grinding and polishing. One top end 1e of the tube may be machined in a sphere with a chamfer at the edge and other end may be machined to form a chamfer or cone at the opening of the through hole.

[0031]

The material for the ferrule may preferably have hardness enough to avoid the ferrule from breaking while fabricating or using it. When the ferrule is protected by a backing body, as shown in Fig. 1, from which an end portion of the ferrule projects, the ferrule is desirable to bear the weight W of 3.5 kg or more which is loaded perpendicular to its axis on the ferrule at a distance of 6.5 mm from its top end.

[0032]

The bending strength  $\sigma$  to be required for the ceramic material is defined as follows;

$$\sigma \geq M/Z = W \times 6.5 / (\pi d^3 / 32) = 1483 \text{ kg/cm}^2$$

where diameter d of a ferrule is 2.5 mm, moment M and sectional area coefficient Z. Therefore, ferrules may be formed of the ceramics having the bending strength of 1500 kg/cm<sup>2</sup>.

[0033]

[Examples]

The examples of the present invention will be explained below.

[0034]

Ferrules, as shown in Fig. 1, were produced of various materials. The ferrules had dimensions of 10.5 mm in length and 2.5 mm in outer diameter, with a through hole 1a of 0.126 mm in inner diameter. 20 samples of each material were prepared. Each sample was kept in a incubator at the temperature of 40°C for 2 hours, then, cooled naturally to be made dry, and thereafter weighed to obtain a weight before testing by a chemical balance (accuracy  $\pm 0.05\text{mg}$ ).

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[0035]

The samples were held at 85°C and relative humidity of 85% for 2000 hours in an incubator, then weighed in the same manner to obtain the weight after testing. The sample having greatest change in weight before and after testing of the 20 samples in each group was selected to calculate the ratio of the weight change with respect to the weight before the testing.

[0036]

The result is shown in Table 1.  $Y_2O_3$ -stabilized zirconia (Sample No. 1) shows a large weight change rate of 0.138 wt%, due to a hydrated layer on the surface. Cordierite and mulite ceramics (Sample Nos. 15 and 25) are very high in weight change rate. This is because of high porosity. It is possible to decrease the change rate below 0.1 wt% by decreasing the porosity of the ceramics into 0.1% or less. Further, the resin materials, Samples Nos. 25 and 26 showed high rate of weight change because of absorbing water.

[0037]

However, the other ceramics were able to be made low in a weight change rate to 0.1% or less.

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[0038]

[Table 1]

No.	Material	weight change (mg)			rate (wt%)
		before	after	differ.	
*1	Y <sub>2</sub> O <sub>3</sub> zirconia	304.31	304.73	0.42	0.138
2	Dy-Ce zirconia	294.21	294.28	0.07	0.024
3	MgO zirconia	289.12	289.18	0.06	0.021
4	CaO zirconia	294.36	294.30	-0.06	-0.020
5	Alumina(Purity 99.7%)	197.85	197.89	0.04	0.020
6	Alumina(Purity 99.0%)	197.73	197.78	0.05	0.025
7	Alumina(Purity 96%)	194.56	194.64	0.08	0.041
8	Alumin+ziO <sub>2</sub> 5mol%	208.45	208.49	0.04	0.019
9	Alumin+ziO <sub>2</sub> 20mol%	223.75	223.71	-0.04	-0.018
10	Alumin+ziO <sub>2</sub> 2.5mol%	205.82	205.89	0.06	0.029
11	nano sructure Alumina+Al <sub>2</sub> B <sub>2</sub> O <sub>6</sub>	167.42	167.47	0.05	0.030
12	Steatite	143.36	143.36	0	0
13	Titania	204.80	204.72	-0.08	-0.039
14	Sapphire	203.64	203.59	-0.08	-0.025
*15	Cordierite	76.80	97.38	20.58	26.79
16	Forsterite	153.62	153.67	0.05	0.033
17	Silicon carbide	163.89	163.94	0.05	0.031
18	Silicon Nitride	163.53	163.49	-0.04	-0.024
19	Aluminum nitride	174.08	174.13	0.05	0.029
20	Cermet	304.38	304.32	-0.06	-0.020
21	Borosilicate glass	120.83	120.82	-0.01	0.008
22	crystalized glass	128.51	128.53	0.02	0.016
23	SiC-Al Composite	158.75	158.78	0.03	0.019
24	Mulite	102.43	109.95	7.52	7.34
*25	PBT resin	77.83	77.90	0.07	0.089
*26	PEI resin	68.61	68.80	0.19	0.277

\* outside the present invention

[0039]

Next, the 20 samples were prepared, by inserting an optical fiber through the ferrule in each ferrule and pressing the ferrule into the backing body, and were held under the condition of 85°C of temperature and 85% of relative humidity conditions for 2000 hours in a incubator, then, comparing optical losses in connecting the fibers between before and after the testing.

[0040]

The result is shown in Table 2. the Samples Nos. 1, 15, and 24-26 are apt to increase in optical loss or be broken when the rate of change in

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weight exceeds 0.1 wt% during the test.

[0041]

On the contrary, the other samples do not almost produce the optical loss, and are assured high useful durability even in use in a high temperature and humidity circumstances. Therefore, The materials which are formed in the condition that the weight change rate is 0.1 wt% or less during testing for 2000 hours at a temperature of at 85°C and relative humidity of 85%, can increase the durability of ferrules which is used in a hot and wet circumstances.

[0042]

[Table 2]

No.	material	connecting loss (dB)	
		before testing	after testing
1	Y <sub>2</sub> O <sub>3</sub> Zirconia	0.18	0.43
15	Cordierite	0.19	broken
24	Mulite	0.22	broken
25	PBT resin	0.17	broken
26	PEI resin	0.19	broken
average otherwise		0.21	0.23

[0043]

[Effect of the Invention]

As noted above, according to the present invention, a ferrule is provided which has a through hole to hold an optical fiber and is formed of a ceramic material having 0.1 wt% or less of a rate of change in weight while the material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours so that a ferrule for connecting an optical fiber which exhibits high durability in use under high-temperature and high-humidity circumstances and low optical transmission loss of the fiber connector for a long time.

[Brief description of the Drawings]

[Fig. 1] shows a side view of a ferrule for an optical fiber connector according of the present invention.

[Description of the Numerals]

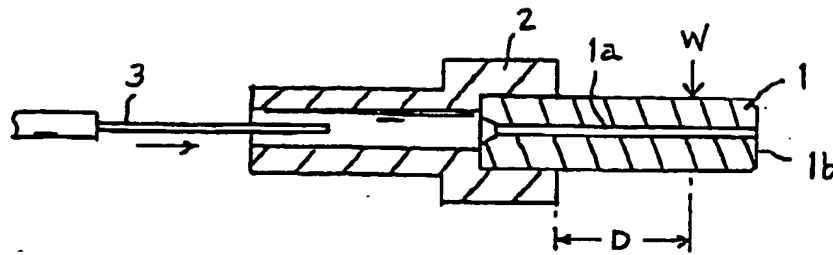
- 1: ferrule
- 1a: through hole
- 1b: end face
- 2: backing body

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**3: optical fiber**

[document] drawings

[Fig. 1]



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[document]      Abstract

[Abstract]

[Problems] A ferrule for an optical fiber connector is provided to exhibits high durability in use under high-temperature and high-humidity circumstances and optical transmission loss of the fiber connector for a long time.

[solution] The ferrule 1 having a through hole 1a to holding an optical fiber 2 therein is formed of a ceramic material having 0.1 wt% or less of a rate of change in weight while the material is held at a temperature of 85°C and relative humidity of 85% for 2000 hours.

[Selected Drawing] Fig. 1